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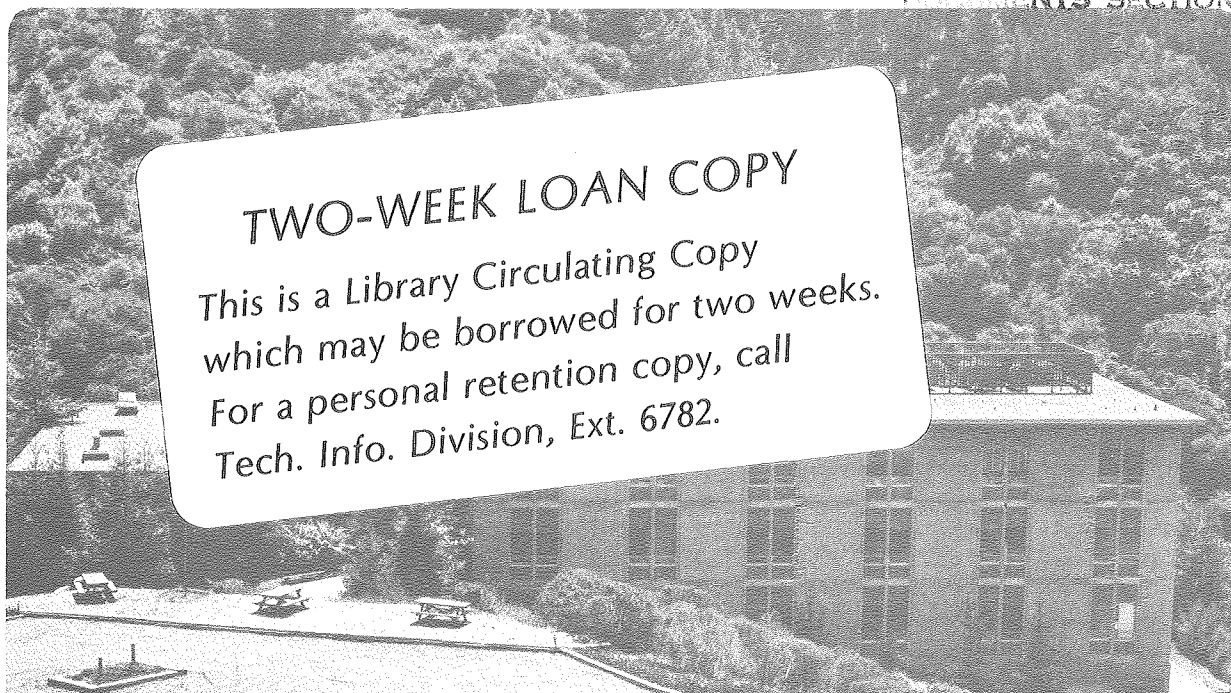
D. Grivas, J.W. Morris, Jr., and T.G. Langdon

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OBSERVATIONS ON THE DIFFERENCES REPORTED IN REGION I
FOR THE SUPERPLASTIC Zn-22% Al EUTECTOIDD. Grivas[†], J. W. Morris, Jr.[†] and T. G. Langdon[‡][†]Department of Materials Science and Mineral Engineering, University of California, Berkeley, and Materials and Molecular Research Division, Lawrence Berkeley Laboratory, Berkeley, California 94720.[‡]Departments of Materials Science and Mechanical Engineering, University of Southern California, Los Angeles, California 90007.

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1. Introduction

The Zn-22% Al eutectoid alloy is a superplastic material which has been used in a number of experimental investigations of superplasticity. However, despite the considerable attention devoted to this metal, the published experimental results fall into two separate categories. These two types of behavior are illustrated schematically in Fig. 1, using the standard logarithmic plot of stress, σ , versus strain rate, $\dot{\epsilon}$, for a superplastic material deforming under steady-state conditions.

In the early experiments on Zn-22% Al by Vaidya *et al.* (1), it was reported that the stress-strain rate relationship was divisible into three distinct regions such that the strain rate sensitivity, m ($= \partial \ln \sigma / \partial \ln \dot{\epsilon} = 1/n$, where n is the stress exponent), was low at high strain rates ($m \approx 0.27$), increased to a higher value at intermediate strain rates ($m \approx 0.5$), and then increased again at very low strain rates ($m \approx 1.1$). These three regions are designated, respectively, III, II, and I in Fig. 1, and these early experiments also showed a corresponding change in the activation energy, Q , from a high value close to lattice self-diffusion in region III to a lower value close to grain boundary diffusion in regions II and I.

Subsequent experiments on Zn-22% Al have tended to confirm the results of Vaidya *et al.* (1) in regions III and II, but they have revealed marked differences in the low stress region I so that the published data divide into two distinct classes. On the one hand, Misro and Mukherjee (2) confirmed the results of Vaidya *et al.* (1) with $m \approx 1.0$ and a low activation energy in region I. Very recently, Arieli *et al.* (3) again obtained $m \approx 1.0$ in region I but with a high activation energy close to lattice self-diffusion due, it was suggested, to a transition from Coble (4) to Nabarro-Herring (5,6) diffusion creep at large grain sizes. On the other hand, Mohamed and Langdon (7) obtained a decrease in m on passing from regions II to I, with $m \approx 0.24$ and a corresponding increase in the activation energy at the lowest strain rates (8). The decrease in m to ~ 0.3 in region I was subsequently confirmed by Grivas (9), and Vale *et al.* (10) confirmed both the decrease in m and the corresponding increase in the activation energy.

There are two obvious discrepancies in the published data for Zn-22% Al at low strain rates.

First, Mohamed and Langdon (7,8), Vale *et al.* (10) and Arieli *et al.* (3) report a high activation energy in region I, whereas Vaidya *et al.* (1) and Misro and Mukherjee (2) report a low activation energy. There appears to be no rational explanation for this dichotomy. However, it is interesting to note that Arieli *et al.* (3) attributed their high activation energy of 95.9 ± 2.1 kJ mol⁻¹ to Nabarro-Herring creep with $m = 1$ when using grain sizes of 1.3 to 3.7 μ m, whereas Vaidya *et al.* (1) obtained $Q = 74.6 \pm 0.8$ kJ mol⁻¹ for grain sizes of 2.3 and 3.6 μ m and

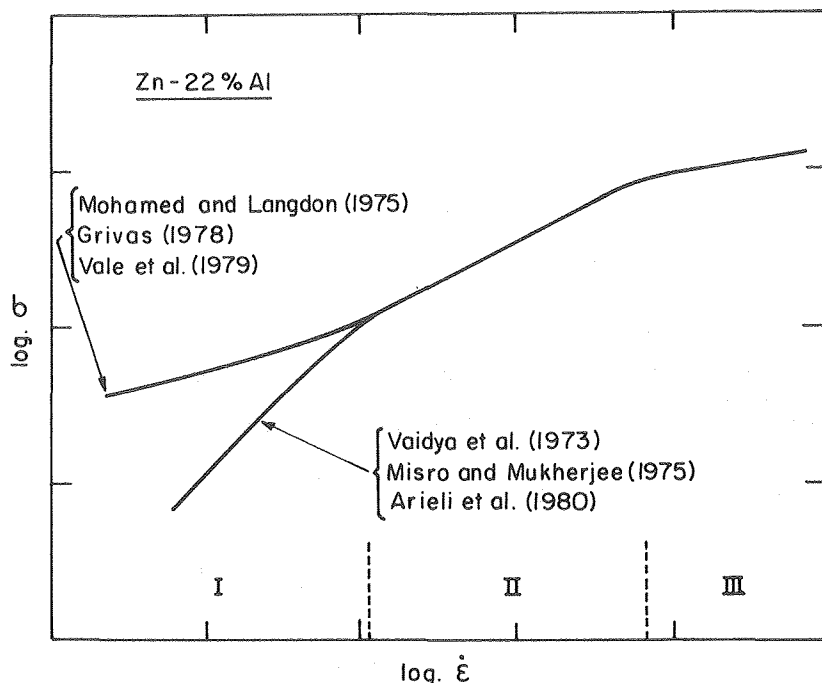


FIG. 1

Schematic illustration of stress versus strain rate in Zn-22% Al, showing the two types of behavior reported in region I.

Misro and Mukherjee (2) obtained $Q = 64.5 \pm 6.3 \text{ kJ mol}^{-1}$ for grain sizes of 0.97 to 2.16 μm . The latter two results were attributed to Coble creep with $m = 1$, although, as documented in Table I, all three sets of data (1-3) refer to identical testing temperatures, T , and very similar ranges of grain size, d .

Second, there is a marked discrepancy between the high value of m obtained in region I by Vaidya *et al.* (1), Misro and Mukherjee (2) and Arieli *et al.* (3) and the low value obtained in the same region by Mohamed and Langdon (7), Grivas (8) and Vale *et al.* (10). This second discrepancy forms the basis for the present paper.

It was first pointed out by Rai and Grant (11), in experiments on Al-33% Cu, that a false "region I" with a low value of m may be observed at low strain rates due to the occurrence of concurrent grain growth during the test. Since the strain rate in region II is approximately proportional to the inverse square of the grain size, any growth of grains on a large scale would tend to be compensated by an increase in the stress level under conditions of constant imposed strain rate. This suggestion was subsequently adopted for Zn-22% Al by Arieli and Mukherjee (12) in an attempt to explain the two types of behavior depicted in Fig. 1. As will be demonstrated in section 2, concurrent grain growth is unable to account for the results obtained in Zn-22% Al when $m \approx 0.3$ in region I. An alternative possibility is presented in section 3 which suggests that the reports of $m \approx 1$ in Zn-22% Al in region I are probably in error.

2. An Examination of Concurrent Grain Growth in Zn-22% Al

Arieli and Mukherjee (12) attempted to incorporate the effect of concurrent grain growth into an analysis of the stress-strain rate results reported by Mohamed *et al.* (7,8). For this analysis, the change in grain size was represented by the expression

$$\frac{\Delta d}{d_0} = \frac{d' - d_0}{d_0} = \frac{Bt^c}{d_0} \quad [1]$$

TABLE I

Experimental Conditions and Values Obtained for Q in Region I when $m \approx 1$

Reference	d (μm)	T (K)	n ($=1/m$)	m	Q (kJ mol^{-1})
Vaidya <i>et al.</i> (1)	2.3, 3.6	448 - 523	0.87	1.1	74.6 ± 0.8
Misro and Mukherjee (2)	0.97 - 2.16	423 - 523	1.0	1.0	64.5 ± 6.3
Arieli <i>et al.</i> (3)	1.3 - 3.7	450 - 525	1.0	1.0	95.9 ± 2.1

where Δd is the change in grain size, d_0 is the initial grain size, d' is the instantaneous grain size, t is the time, c is the time exponent, and B is a material constant. From equation [1], it follows that

$$\log \frac{\Delta d}{d_0} = \log B + c \log \left(\frac{\dot{\epsilon}}{\dot{\epsilon}_0} \right) - \log d_0 \quad [2]$$

where ϵ is the strain and $\dot{\epsilon}$ is the strain rate.

In order to make use of equation [2], Arieli and Mukherjee (12) replotted some grain growth results reported earlier for Zn-22% Al by Mohamed *et al.* (13). The data were logarithmically plotted as $\Delta d/d_0$ versus $\dot{\epsilon}$ [Fig. 3 of reference (12)] and it was erroneously assumed that the strain, ϵ , in equation [2] was constant. In fact, the grain growth data were taken from measurements performed on a series of specimens after fracture, so that there were very significant differences in the strains associated with each separate measurement.

Two points should be noted concerning concurrent grain growth in the Zn-22% Al alloy. First, a preferred procedure for plotting the grain growth data is in accordance with the empirical relationship developed for grain growth during isothermal annealing tests (14,15):

$$d = Kt^q \quad [3]$$

where K and q are constants and $q \leq 0.5$. The result is shown in Fig. 2 for Zn-22% Al tested at 503 K with an initial grain size of 2.5 μm : all datum points are taken from Mohamed *et al.* (13), and the single point designated without a stress refers to a specimen held at 503 K for the time of the longest test ($\sim 3 \times 10^5$ s). From Fig. 2, it is estimated that $q \approx 0.04$, so that grain growth during testing is clearly of only very minor significance in Zn-22% Al. Second, it has been demonstrated already by calculation (13) that the very small amount of grain growth observed in Zn-22% Al, at least in the experiments conducted at 503 K, is unable to account for the observed transition in the mechanical data to region I and $m \approx 0.3$.

Based on the available evidence, it is clear that the analysis and subsequent conclusions presented by Arieli and Mukherjee (12) are in error. Furthermore, additional support for this viewpoint may be found in the recent work of Vale *et al.* (10), where there is a clearly defined region I with $m \approx 0.32$ and yet, according to the authors, their specimens "exhibited negligible grain growth."

3. An Examination of the Role of Primary Creep

The preceding discussion demonstrates that it is not feasible to attribute the low values of m obtained in region I in some experiments (7,9,10) to the advent of concurrent grain growth. It is therefore necessary to now develop an alternative explanation for this apparent dichotomy.

It is possible, and indeed seems probable, that the high values of m (~ 1.0) observed in some investigations at low strain rates (1-3) are due to the occurrence of a substantial primary stage of creep. If the strain rate was measured in a creep experiment before the true steady-state situation, the recorded value of $\dot{\epsilon}$ would be erroneously high and may give rise to a false "region I" with a high value of m . An examination of the three sets of experiments reporting $m \approx 1$ at low strain rates shows inconsistencies on this point: Vaidya *et al.* (1) reported that steady-state creep in region I was "always preceded by reasonably long transients" (although it is interesting to note that this observation is inconsistent with their proposal for Coble creep

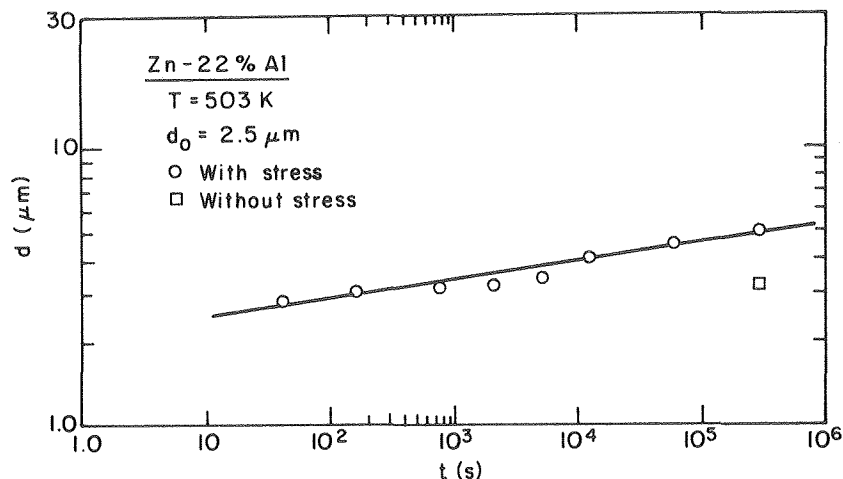


FIG. 2

Measured grain size versus time for Zn-22% Al tested at 503 K.

in this region), Misro and Mukherjee (2) stated that the creep curves in region I "did not have any primary or transient stage," and Arieli *et al.* (3) show evidence of a primary stage in a plot of strain versus time (their Fig. 6).

The difficulty associated with the presence of primary creep, especially at low strain rates, may be illustrated very easily. Figure 3 shows a typical creep curve for a specimen of Zn-22% Al, having an initial grain size of $1.9 \mu\text{m}$, tested in double shear at 523 K at a shear stress, τ , of 0.1 MPa in region I. The curve plots shear strain, γ , against time, and shows the pronounced primary stage preceding steady-state conditions. The steady-state shear strain rate was estimated from the datum points at $\gamma > 0.10$ as $\dot{\gamma} = 1.1 \times 10^{-6} \text{ s}^{-1}$. However, it is clear that an erroneously high strain rate would be recorded at a lower shear strain of, for example, 0.05. To illustrate this point, Fig. 4 shows the datum points obtained from an experiment in which the stress was changed periodically and the values of $\dot{\gamma}$ were estimated at fixed shear strains of $\gamma = 0.05$ after each stress change. In this case, which does not relate to true steady-state conditions, there is an apparent transition from $n \approx 3$ ($m \approx 0.3$) in "region II" to a false $n \approx 1$ ($m \approx 1$) in "region I." It seems probable that this type of error would occur more easily under conditions where it is anticipated that diffusion creep prevails and there is little or no primary stage.

The conclusions from this work are therefore three-fold. First, whereas the three sets of experiments (7,9,10) showing $m \approx 0.3$ at low stress levels in region I in Zn-22% Al are mutually consistent, there are at least two major inconsistencies in the three experiments reporting $m \approx 1.0$ at the same low stresses. These points of inconsistency are (i) wide variations in the reported values for the activation energies, as documented in Table I, which cannot be attributed to differences in grain size or other testing conditions, and (ii) disagreement on the shape of the creep curves in region I, with reports of "long transients" (1) and no transient stage (2). Second, there is a gross error in the analysis of concurrent grain growth in Zn-22% Al by Arieli and Mukherjee (12), and their conclusion that grain growth accounts for region I with $m \approx 0.3$ is unfounded. Third, the three reports of $m \approx 1$ in region I (1-3) almost certainly arise because of a failure to accurately account for the primary stage of creep, as demonstrated in Fig. 4.

Finally, it should be noted that the important conclusion from this analysis, that the reports of $m \approx 1$ in region I (1-3) are incorrect, arises solely from a detailed examination of the various sets of data. However, there are also various other experimental results on Zn-22% Al which make it difficult to sustain a direct transition from $m \approx 0.5$ in region II to $m \approx 1.0$ in region I, including a decrease in ductility at low strain rates (16), evidence for neck formation in region I (17), and a decrease in the contribution from grain boundary sliding at very low strain rates (18).

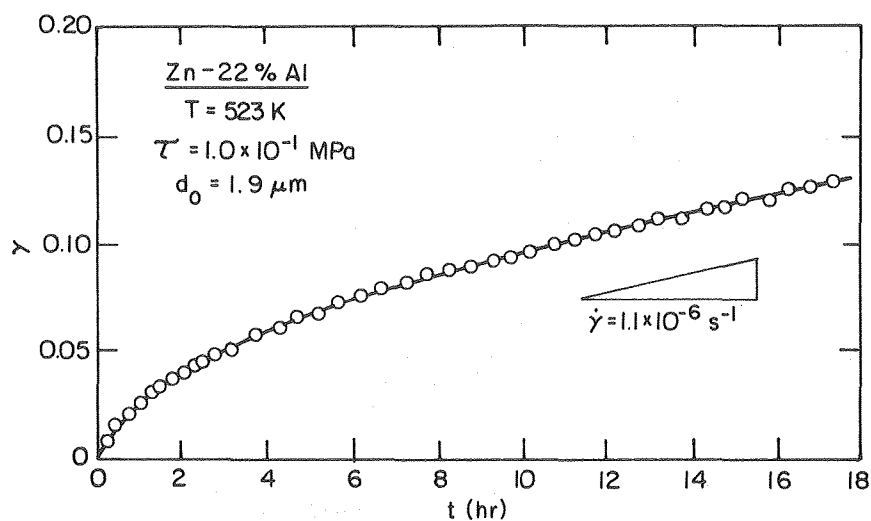


FIG. 3

Creep curve for Zn-22% Al tested in double shear at 523 K in region I.

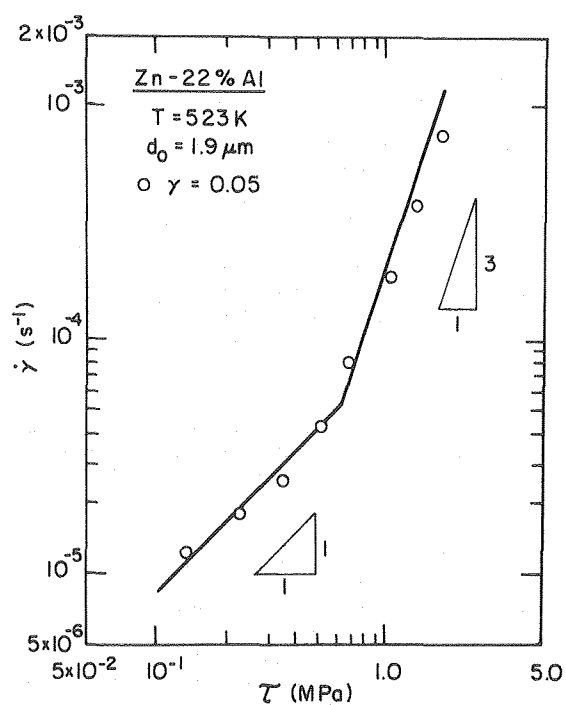


FIG. 4

Apparent variation of shear strain rate with stress when readings are taken at fixed shear strains of 0.05 rather than under true steady-state conditions.

4. Summary and Conclusions

1. There are three consistent reports (7,9,10) of a strain rate sensitivity, m , of ~ 0.3 in Zn-22% Al at low strain rates in region I, whereas the three reports of $m \approx 1.0$ in region I (1-3) show major inconsistencies.

2. Concurrent grain growth is unable to account for the reports of $m \approx 0.3$ in region I, and the analysis (12) purporting to demonstrate the influence of grain growth in Zn-22% Al is in error.

3. It is concluded that the reports of $m \approx 0.3$ in region I are correct, and the observations of $m \approx 1.0$ are almost certainly due to a failure to accurately account for the primary stage of creep.

Acknowledgments

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ADDENDUM:

This paper discusses the different types of creep behavior reported for the steady-state deformation of the superplastic Zn-22% Al eutectoid alloy at low stress levels. Arieli and Mukherjee (19) have discussed the paper. The purpose of this addendum is to summarize the various views and present a brief response.

To place the following discussion in its proper perspective, we re-emphasize the fact that all existing theories of creep behavior predict that at sufficiently low stresses the steady-state creep will be diffusion-controlled and will follow a power-law with a strain rate exponent of $m = 1.0$. The question before us is whether this diffusional creep mechanism sets the lower bound on superplastic deformation in typical samples of Zn-22% Al, or whether another type of creep behavior intervenes. Arieli and Mukherjee (19) claim a direct transition from superplastic to diffusional creep and cite three sets of experiments (1-3) which purport to show a break in the power-law creep curve from a superplastic region II ($m \approx 0.5$) to a diffusion-controlled region I ($m \approx 1.0$). Both we (7,9) and Vale *et al.* (10) studied the behavior of similar samples over the same range of creep conditions and found that, on the contrary, superplastic deformation is limited by a transition to a region I of power-law creep which may be dislocation-controlled

TABLE II

Thermomechanical Treatments Used for Specimens Showing $m \approx 1.0$ in Region I

<u>Material Source</u>	<u>Rolling condition</u>	<u>Solution Treatment</u>	<u>Annealing condition</u>	<u>Reference</u>
(Not stated)	At 593 K to 2.22 cm	In argon at 648 K for 15 hr; quench to ice water	At 538 K for periods from 6 hr to 5 weeks	Vaidya <u>et al.</u> (1)
Kaiser Aluminum & Chemical Corp. [†]	At 617 K to 0.32 cm [†]	In argon at 648 K for 16 hr; quench to CaCl ₂ + ice water	At 528 K for periods from 10 min to 40 hr	Misro and Mukherjee (2)
Kaiser Aluminum & Chemical Corp.	At 617 K to 0.32 cm	In argon at 648 K for 16 hr; quench to CaCl ₂ + ice water	At 528 K for periods from 10 min to 40 hr	Arieli <u>et al.</u> (3)

[†]Details obtained from Misro (20).

and has $m \approx 0.3$. While it is always possible that some subtle consistent difference in the experimental materials or testing procedure is responsible for the discrepancy, a review of the relevant experimental papers suggests that this is unlikely. We argue with Arieli and Mukherjee (19) that it is much more likely that one of the two sets of observations is in error. We examine the two possibilities in turn.

A. On the possibility that the experiments reporting $m \approx 1.0$ are in error.

We believe that the reports of a region I with slope $m \approx 1.0$ are spurious. Our opinion is based on the results of experiments we believe to have been carefully conducted (7,9,10) and on two additional considerations: (1) the internal inconsistency of the data supporting $m \approx 1.0$ and (2) the demonstrated possibility of obtaining $m \approx 1.0$ when the experiment is done improperly.

(1) There are both quantitative and qualitative inconsistencies between the three sets of experiments (1-3) purporting to show $m \approx 1.0$ in region I. The most important of these concern the activation energy for creep and the presence of primary creep. Misro and Mukherjee (2) report an activation energy of 64.5 kJ mol^{-1} while Arieli et al. (3) report a much higher activation energy of 95.9 kJ mol^{-1} . Misro and Mukherjee (2) report that the creep curves "did not have any primary stage" whereas Arieli et al. (3) report "clear evidence of a primary stage." As documented in Table I [and accepted by Arieli and Mukherjee (19)] these differences cannot be attributed to testing conditions since the temperatures, grain sizes, and testing procedures were very similar in all three experiments.

In their response to this paper, Arieli and Mukherjee (19) address these problems by arguing that the inconsistency in the activation energies is due to "microstructural and phenomenological factors which affect the transition from one creep mechanism to another" and that the difference in primary creep behavior occurs because "starting materials in various investigations had different initial substructures." These arguments are undeveloped and unsupported. The substructural factors to which they allude are neither specified precisely nor shown in the context of this or other experimentation to give rise to the experimental discrepancies they are trying to explain.

Our review of the work cited by Arieli and Mukherjee leads to precisely the opposite conclusion: that the initial substructures should have been remarkably similar in all three experiments. As documented in Table II, all three sets of specimens were given very similar thermomechanical treatments prior to testing. In fact, the experiments of Misro and Mukherjee (2) and Arieli et al. (3) used material from the same source prepared in an identical manner.

We note by contrast the mutual consistency of the three reports of a transition to $m \approx 0.3$ in region I (7,9,10). These three experiments were done in different laboratories by different investigators using starting materials from different sources with various initial preparations. Moreover, Mohamed and Langdon (7) performed tests on both a laboratory material and a commercial alloy and noted that "the results obtained on the materials from these two different sources are essentially identical."

(2) Grivas (9) has shown that the conclusion of $m \approx 1.0$ in region I can be reached as the natural outcome of creep experiments which are improperly and impatiently conducted. His results

are discussed in detail in section three of this paper and derive from the extensive primary creep in region I (Fig. 3). Stress change experiments in which strain rates are recorded at low strain intervals ($\gamma = 0.05$) give rise to an erroneous "region I" with $m \approx 1.0$ (Fig. 4).

Arieli and Mukherjee (19) counter this argument by claiming that they were aware of primary creep and that, hence, "all of the data used in [Arieli *et al.* (3)] were collected at $\gamma \geq 0.1$." To verify this statement, we reexamined the paper by Arieli *et al.* (3). The only place in that paper where data are presented from which the strain prior to the strain rate measurement could be inferred is in their Fig. 6 (3). This figure shows a stress change experiment in region I in which the "stress was increased after apparent steady state had been reached at a lower stress." In fact, the change in stress was made at $\gamma \approx 0.02$, which is not only well below the claimed strain of $\gamma \geq 0.1$ but is also below the strain interval of $\gamma = 0.05$ at which Grivas obtained the spurious result of $m \approx 1.0$. Since the detailed nature of Fig. 6 (3) makes a presentation error unlikely, we are forced to conclude that either Arieli and Mukherjee misremember how they conducted these experiments or that they chose to present, as their only experimental example, a representation of an atypical experiment which they themselves knew to be spurious. Given the consistency of Grivas' results with those which would have been obtained if the experiments had actually been conducted as diagrammed in Fig. 6 (3), we suspect that they misremember their experimental procedure.

B. On the possibility that the experiments reporting $m \approx 0.3$ are in error.

The argument presented by Arieli and Mukherjee (19) against the experiments reporting $m \approx 0.3$ in region I concerns the possible influence of concurrent grain growth on the measured strain rate exponent. The issue of grain growth is discussed in some detail in the body of this paper. By way of addendum, we simply wish to point out that if Arieli and Mukherjee are right the correctness of their position will be manifest in at least two types of experiment: (1) the exponent m will not be near to 0.3 in experiments involving negligible grain growth, and (2) the value of the exponent m will depend on the order in which the data are taken; the value of m found from a series of tests on a single sample at progressively increasing loads will necessarily differ from that inferred from a series of tests on an identical sample using progressively decreasing loads. Both types of experiment have been conducted.

(1) Regarding tests at constant grain size, we refer to the work of Vale *et al.* (10) and specifically to their discussion of the discrepancy between the results of Mohamed and Langdon (7) and Arieli and Mukherjee (12): "The differences cannot be explained by grain growth during mechanical tests in [the work of Mohamed and Langdon], as suggested by Arieli and Mukherjee, because our samples exhibited negligible grain growth and yet gave results similar to Mohamed and Langdon."

(2) Regarding the possible influence of the testing sequence, we refer to the work of Grivas (9). By alternately increasing and decreasing the load in region I, he found that it was possible to cycle a single specimen along a single steady-state line having an exponent of $m \approx 0.3$. Such a result is clearly impossible if the strain rate exponent itself depends on grain growth during testing.

We therefore restate our conclusion that the consistent observations (7,9,10) of $m \approx 0.3$ in region I of the steady-state creep deformation of Zn-22% Al cannot be attributed to grain growth during testing and provide a valid representation of the creep behavior of the material. The reports of $m \approx 1.0$ (1-3) derive from experiments whose results are mutually inconsistent and offer a strain rate exponent which has also been found as the spurious result of experiments which were intentionally done in an improper way. We believe these reports are wrong.

This conclusion leaves open the question of whether a true diffusion-controlled creep bounds region I on the low stress side, as we expect theoretically. Some evidence for this transition was presented by Vale *et al.* (10), and they refer to a region 0. Both Langdon and Mohamed (21) and Grivas (9) examined the issue theoretically with reference to their own testing conditions, and concluded that a well-articulated diffusional creep behavior would only be found at stresses below the lowest values used experimentally.

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